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(54) **Terminating post cure of silicone elastomers with amino acid esters**

(57) A method of thickening organic solvents and silicone fluids with elastomers involves reacting (A) an =Si-H containing polysiloxane with (B) an alkene such as an alpha, omega-diene; conducting the reaction in the presence of catalyst (C) and (D) a solvent; continuing the reaction until an elastomer is formed by crosslinking and addition of =Si-H across double bonds

in the alkene; adding additional solvent and a post cure terminating agent (E) to said elastomer; and subjecting the solvent, post cure terminating agent and elastomer to shear force until a paste is formed. The post cure terminating agent (E) is an amino acid ester, preferably a sulfur containing amino acid ester, such as methionine methyl ester, methionine ethyl ester, cysteine methyl ester, cysteine ethyl ester and cystine dimethyl ester.

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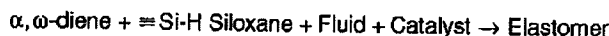
Description

[0001] This invention is directed to methods for the termination of post cure occurring in the thickening of silicone fluids and organic solvents with silicone elastomers.

5 [0002] In US-A 5,654,362, silicone elastomers are used in the thickening of silicone fluids and organic solvents. These elastomers are formed by a hydrosilylation reaction between a multifunctional $\equiv\text{SiH}$ siloxane and an alkene, preferably α,ω -diene.

[0003] According to the above patent, one process that is used in making a silicone elastomer suitable in thickening a low viscosity silicone fluid or organic solvent is:

10 Step 1 - Gelation



15 Step 2 - Shear & Swell



20 [0004] When a crosslinked network is formed in such a reaction, we have determined that steric hindrance of the crosslinked structure prevents the reaction from reaching completion. This is due to the fact that a small amount of residual functionality will remain even after long reaction times and that unreacted functionalities will tend to meet each other when the elastomer is sheared and swollen. We have termed this phenomenon as "post cure". Because residual reactivity causes smooth pasty products to further gel, post cure should be eliminated to maintain a finer appearance and a more flowable rheology of the final paste product.

25 [0005] This is easily achieved, according to this invention, by deactivating the catalyst with an amino acid ester containing post cure terminating agent, most preferably a sulfur containing amino acid ester.

[0006] We have discovered that the post cure caused by residual crosslinking hydrosilylation reactions, which typically occur in the preparation of silicone elastomers, are conveniently terminated by introducing a strong platinum group metal complexing ligand to deactivate the catalyst. The aforementioned ligand is preferably an amino acid ester; and most preferably, it is a sulfur containing amino acid ester. In our claimed method, the resulting product is not contaminated by any toxic ingredients. This is an important feature, advantage and benefit, when such products are intended for use in the personal care or health care arenas.

30 [0007] Accordingly, our inventive method for termination of post cure involves introducing a small amount (one equivalent or more) of a strong platinum group metal complexing ligand, such as an amino acid ester. Especially preferred ligands of our invention are sulfur containing amino acid esters, such as methionine methyl ester $\text{CH}_3\text{SCH}_2\text{CH}_2\text{CH}(\text{NH}_2)\text{COOCH}_3$, methionine ethyl ester $\text{CH}_3\text{SCH}_2\text{CH}_2\text{CH}(\text{NH}_2)\text{COOC}_2\text{H}_5$, cysteine methyl ester $\text{HSCH}_2\text{CH}(\text{NH}_2)\text{COOCH}_3$, cysteine ethyl ester $\text{HSCH}_2\text{CH}(\text{NH}_2)\text{COOC}_2\text{H}_5$ and cystine dimethyl ester $[-\text{SCH}_2\text{CH}(\text{NH}_2)\text{COOCH}_3]_2$.

35 [0008] As a result, we have found that the catalyst is readily deactivated and that further reaction is impaired in the final product. Our method is not only effective but it is also less hazardous since naturally occurring amino acid derivatives are employed. This is an important feature when the ultimate product is intended for application in the personal care or health care arenas.

40 [0009] Because amine and sulfide functionality are good binding ligands for platinum group metals, amino acid esters have in their free base form amino groups available to complex such catalysts in a hydrosilylation reaction. Even more importantly, however, when the amino acid esters contain sulfur, said sulfide deactivates the catalysts even more effectively.

45 [0010] For our invention, it is preferred that the amino acids not be in zwitterionic form. Rather, they should be derivatized to other forms, such as an ester, so they are more compatible in silicone and organic systems. Additionally, the amino acid derivatives should be in free base form and not in the ammonium salt form; so they are more compatible in silicone and organic systems and they have amino groups available to complex the catalyst. Alternatively, the amino acid derivatives can be converted to a free base form by a simple acid-base reaction after being mixed in the system.

50 [0011] Our invention is useful in any process involving silicone elastomers prepared by a crosslinking reaction between (A) $\equiv\text{Si-H}$ containing polysiloxanes and (B) an alkene such as an alpha, omega-diene, in the presence of a platinum group metal catalyst (C) and (D) a solvent. Such elastomers are typically swollen with low molecular weight polysiloxanes under a shear force.

55 [0012] Usually, the $\equiv\text{Si-H}$ containing polysiloxane (A) is a polymer represented by the formula $\text{R}_3\text{SiO}(\text{R}'_2\text{SiO})_a(\text{R}''\text{HSiO})_b\text{SiR}_3$ designated herein as type A¹ and polymers of the formula $\text{HR}_2\text{SiO}(\text{R}'_2\text{SiO})_c\text{SiR}_2\text{H}$ or $\text{HR}_2\text{SiO}(\text{R}'_2\text{SiO})_a(\text{R}''\text{HSiO})_b\text{SiR}_2\text{H}$ designated herein as type A². In these formulas, R, R' and R'', are alkyl groups with 1-6 carbon atoms;

a is 0-250; b is 1-250 and c is 0-250. The molar ratio of compounds A²:A¹ is 0-20, preferably 0-5. In preferred embodiments, compounds of types A¹ and A² are used in the reaction; however, it is also possible to conduct the reaction using only compounds of type A¹.

[0013] The term alkene (B) includes, and most preferably comprises, an alpha, omega-diene of the formula CH₂=CH(CH₂)_xCH=CH₂ where x is 1-20. Representative alpha, omega-dienes are 1,4-pentadiene; 1,5-hexadiene; 1,6-heptadiene; 1,7-octadiene; 1,8-nonadiene; 1,9-decadiene; 1,11-dodecadiene; 1,13-tetradecadiene and 1,19-eicosadiene.

[0014] The term alkene is also intended to include siloxane monomers or polymers containing two or more terminal alkenyl groups; two or more pendant alkenyl groups; or two or more terminal and pendant groups. One suitable siloxane, for example, is tetramethyldivinylidisiloxane.

[0015] These addition and crosslinking reactions require a catalyst (C) to effect reaction between the =SiH containing polysiloxane and the alpha, omega-diene. Suitable catalysts are the noble metals and, more particularly, the platinum group metals. Such metal catalysts are more completely described in US Patent 3,923,705. A preferred platinum catalyst is Karstedt's catalyst which is a platinum divinyl tetramethyl disiloxane complex, typically containing one weight percent of platinum, carried in a polydimethylsiloxane fluid or in an organic solvent such as toluene.

[0016] Another preferred platinum catalyst is a reaction product of chloroplatinic acid and an organosilicon compound containing terminal aliphatic unsaturation. It is further described in US Patent 3,419,593. These noble metal catalysts are normally used in amounts from 0.00001-0.5 part, per 100 weight parts of the =SiH containing polysiloxane. Preferably 0.00001-0.02 part and, most preferably, 0.00001-0.002 part, are used for this invention.

[0017] The solvents (D) employed herein include organic compounds and silicone fluids of various types. By "solvent", we mean (i) organic compounds, (ii) compounds containing a silicon atom, (iii) mixtures of organic compounds, (iv) mixtures of compounds containing a silicon atom, or (v) mixtures of organic compounds and compounds containing a silicon atom; used on an industrial scale to dissolve, suspend or change the physical properties of other materials.

[0018] The term oil or fluid includes compounds containing a silicone atom, such as (i) low molecular weight linear and cyclic volatile methyl siloxanes, (ii) low molecular weight linear and cyclic volatile and non-volatile alkyl and aryl siloxanes and (iii) low molecular weight linear and cyclic functional siloxanes. Most preferred, however, are low molecular weight linear and cyclic volatile methyl siloxanes (VMS).

[0019] VMS compounds correspond to the average unit formula (CH₃)_aSiO_{(4-a)/2} in which a has an average value of two to three. These compounds contain siloxane units joined by =Si-O-Si= bonds. Representative units are monofunctional "M" units (CH₃)₃SiO_{1/2} and difunctional "D" units (CH₃)₂SiO_{2/2}.

[0020] The presence of trifunctional "T" units CH₃SiO_{3/2} results in the formation of branched linear or cyclic VMS. The presence of tetrafunctional "Q" units SiO_{4/2} results in the formation of branched linear or cyclic VMS.

[0021] Linear VMS have the formula

(CH₃)₃SiO{(CH₃)₂SiO}_ySi(CH₃)₃. The value of y is 0-5. Cyclic VMS have the formula {(CH₃)₂SiO}_z. The value of z is 3-6. Preferably, these VMS have a boiling point less than 250°C. and viscosities of 0.65-5.0 centistoke (mm²/s).

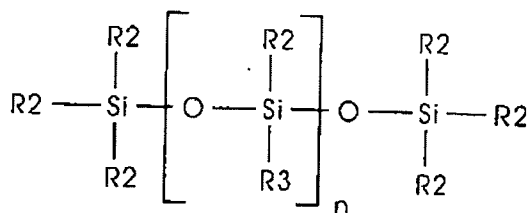
[0022] Representative linear VMS are hexamethyldisiloxane (MM) with a boiling point of 100°C., viscosity of 0.65 mm²/s and formula Me₃SiOSiMe₃; octamethyltrisiloxane (MDM) with a boiling point of 152°C., viscosity of 1.04 mm²/s and formula Me₃SiOMe₂SiOSiMe₃; decamethyltetrasiloxane (MD₂M) with a boiling point of 194°C., viscosity of 1.53 mm²/s and formula Me₃SiO(Me₂SiO)₂SiMe₃; dodecamethylpentasiloxane (MD₃M) with a boiling point of 229°C., viscosity of 2.06 mm²/s and formula Me₃SiO(Me₂SiO)₃SiMe₃; tetradecamethylhexasiloxane (MD₄M) with a boiling point of 245°C., viscosity of 2.63 mm²/s and formula Me₃SiO(Me₂SiO)₄SiMe₃; and hexadecamethylheptasiloxane (MD₅M) with a boiling point of 270°C., viscosity of 3.24 mm²/s and formula Me₃SiO(Me₂SiO)₅SiMe₃.

[0023] Representative cyclic VMS are hexamethylcyclotrisiloxane (D₃) a solid with a boiling point of 134 °C and formula {(Me₂)SiO}₃; octamethylcyclotetrasiloxane (D₄) with a boiling point of 176°C., viscosity of 2.3 mm²/s and formula {(Me₂)SiO}₄; decamethylcyclopentasiloxane (D₅) with a boiling point of 210°C., viscosity of 3.87 mm²/s and formula {(Me₂)SiO}₅; and dodecamethylcyclohexasiloxane (D₆) with a boiling point of 245°C., viscosity of 6.62 mm²/s and formula {(Me₂)SiO}₆.

[0024] Representative branched VMS are heptamethyl-3-((trimethylsilyl)oxy)trisiloxane (M₃T) with a boiling point of 192°C., viscosity of 1.57 mm²/s and formula C₁₀H₃₀O₃Si₄; hexamethyl-3,3-bis((trimethylsilyl)oxy)trisiloxane (M₄Q) with a boiling point of 222°C., viscosity of 2.86 mm²/s and formula C₁₂H₃₆O₄Si₅; and pentamethyl ((trimethylsilyl)oxy) cyclotrisiloxane (MD₃) with the formula C₈H₂₄O₄Si₄.

[0025] Our process also includes the use of low molecular weight linear and cyclic volatile and non-volatile alkyl and aryl siloxanes represented respectively by the formulas R₃SiO(R₂SiO)_ySiR₃ and (R₂SiO)_z. R is an alkyl group of 1-6 carbon atoms or an aryl group such as phenyl. The value of y is 0-80, preferably 0-20. The value of z is 0-9, preferably 4-6. These polysiloxanes have a viscosity generally in the range of 1-100 centistoke (mm²/s).

[0026] Other representative low molecular weight non-volatile polysiloxanes have the general structure:



¹⁰ where n has a value to provide polymers with a viscosity in the range of 100-1,000 centistoke (mm²/s).

[0027] R2 and R3 are alkyl radicals of 1-20 carbon atoms or an aryl group such as phenyl. Typically, the value of n is about 80-375. Illustrative polysiloxanes are polydimethylsiloxane, polydiethylsiloxane, polymethylethylsiloxane, polymethylphenylsiloxane and polydiphenylsiloxane.

15 **[0028]** Low molecular weight functional polysiloxanes useful herein are represented by acrylamide functional siloxane fluids, acrylate functional siloxane fluids, amide functional siloxane fluids, amino functional siloxane fluids, carbinol functional siloxane fluids, carboxy functional siloxane fluids, chloroalkyl functional siloxane fluids, epoxy functional siloxane fluids, glycol functional siloxane fluids, ketal functional siloxane fluids, mercapto functional siloxane fluids, methyl ester functional siloxane fluids, perfluoro functional siloxane fluids and silanol functional siloxanes.

20 **[0029]** Our invention is not limited to swelling silicone elastomers with only low molecular weight polysiloxanes. Other types of solvents, such as organic compounds, can swell the silicone elastomer. Thus, a single solvent or a mixture of solvents may be used.

[0030] In general, the organic compounds are aromatic hydrocarbons, aliphatic hydrocarbons, alcohols, aldehydes, ketones, amines, esters, ethers, glycols, glycol ethers, alkyl halides or aromatic halides. Representative of some common organic solvents are alcohols such as methanol, ethanol, 1-propanol, cyclohexanol, benzyl alcohol, 2-octanol, ethylene glycol, propylene glycol and glycerol; aliphatic hydrocarbons such as pentane, cyclohexane, heptane, Varnish Makers and Painters (VM&P) naphtha and mineral spirits; alkyl halides such as chloroform, carbon tetrachloride, perchloroethylene, ethyl chloride and chlorobenzene; amines such as isopropylamine, cyclohexylamine, ethanolamine and diethanolamine; aromatic hydrocarbons such as benzene, toluene, ethylbenzene and xylene; esters such as ethyl acetate, isopropyl acetate, ethyl acetoacetate, amyl acetate, isobutyl isobutyrate and benzyl acetate; ethers such as ethyl ether, n-butyl ether, tetrahydrofuran and 1,4-dioxane; glycol ethers such as ethylene glycol monomethyl ether, ethylene glycol monomethyl ether acetate, diethylene glycol monobutyl ether and propylene glycol monophenyl ether; ketones such as acetone, methyl ethyl ketone, cyclohexanone, diacetone alcohol, methyl amyl ketone and diisobutyl ketone; petroleum hydrocarbons such as mineral oil, gasoline, naphtha, kerosene, gas oil, heavy oil and crude oil; lubricating oils such as spindle oil and turbine oil; and fatty oils such as corn oil, soybean oil, olive oil, rape seed oil, cotton seed oil, sardine oil, herring oil and whale oil.

[0031] "Other" miscellaneous organic solvents can also be used, such as acetonitrile, nitromethane, dimethylformamide, propylene oxide, trioctyl phosphate, butyrolactone, furfural, pine oil, turpentine and m-cresol.

[0032] Further encompassed by the term solvent are volatile flavoring agents such as oil of wintergreen; peppermint oil; spearmint oil; menthol; vanilla; cinnamon oil; clove oil; bay oil; anise oil; eucalyptus oil; thyme oil; cedar leaf oil; oil of nutmeg; oil of sage; cassia oil; cocoa; licorice; high fructose corn syrup; citrus oils such as lemon, orange, lime and grapefruit; fruit essences such as apple, pear, peach, grape, strawberry, raspberry, cherry, plum, pineapple and apricot; and other useful flavoring agents including aldehydes and esters such as cinnamyl acetate, cinnamaldehyde, eugenyl formate, p-methylanisole, acetaldehyde, benzaldehyde, anisic aldehyde, citral, neral, decanal, vanillin, tolyl aldehyde, 2,6-dimethyloctanal and 2-ethyl butyraldehyde.

45 **[0033]** In addition, solvent also includes volatile fragrances such as natural products and perfume oils. Some representative natural products and perfume oils are ambergris, benzoin, civet, clove, leaf oil, jasmine, mate', mimosa, musk, myrrh, orris, sandalwood oil and vetiver oil; aroma chemicals such as amyl salicylate, amyl cinnamic aldehyde, benzyl acetate, citronellol, coumarin, geraniol, isobornyl acetate, ambrette and terpinyl acetate; and the various classic family
50 perfume oils such as the floral bouquet family, the oriental family, the chypre family, the woody family, the citrus family, the canoe family, the leather family, the spice family and the herbal family.

[0034] Our basic process is a matter of combining the =SiH containing polysiloxane (A), the alkene (B), the solvent (D) and the catalyst (C); and then mixing these ingredients at room temperature until an elastomer is formed.

[0035] Additional amounts of silicone fluids or solvents are then added to the elastomer and the resulting mixture is subjected to shear force to form a paste. Any type of mixing and shearing equipment may be used to perform these steps such as a batch mixer, planetary mixer, single or multiple screw extruder, dynamic or static mixer, colloid mill, homogenizer, sonolator or any combination thereof.

[0036] Typically, our process uses approximately a 1:1 molar ratio of $\equiv\text{Si-H}$ containing polysiloxane and alkene, such as alpha, omega-diene. Useful products may also be prepared by conducting the above process with an excess of

either the =Si-H containing polysiloxane or the alkene but this is a less efficient use of these components. The remainder of our claimed composition comprises silicone fluids or other solvents, in amounts generally within the range of 65-98 percent by weight of the composition and preferably 80-98 percent by weight.

[0037] An essential feature of this invention is adding one equivalent or more of a post cure terminating agent (E) to deactivate the platinum catalyst (C), at or during the shear and swell step.

[0038] The silicone elastomer, silicone gel and silicone paste compositions of our invention have particular value in the personal care arena. Because of the unique volatility characteristics of the VMS component of these compositions, they can be used alone or blended with other cosmetic fluids, to form a variety of over-the-counter (OTC) personal care products.

[0039] Thus, they are useful as carriers in antiperspirants and deodorants, since they leave a dry feel and do not cool the skin upon evaporation. They are lubricious and will improve the properties of skin creams, skin care lotions, moisturizers, facial treatments such as acne or wrinkle removers, personal and facial cleansers, bath oils, perfumes, colognes, sachets, sunscreens, pre-shave and after-shave lotions, liquid soaps, shaving soaps and shaving lathers. They can be used in hair shampoos, hair conditioners, hair sprays, mousses, permanents, depilatories and cuticle coats, to enhance gloss and drying time and provide conditioning benefits.

[0040] In cosmetics, they will readily function as leveling and spreading agents for pigments in make-ups, color cosmetics, foundations, blushes, lipsticks, lip balms, eyeliners, mascaras, oil removers, color cosmetic removers and powders. They are useful as delivery systems for oil and water soluble substances such as vitamins. When incorporated into sticks, gels, lotions, aerosols and roll-ons, the compositions impart a dry, silky-smooth, payout.

[0041] In addition, the claimed compositions exhibit a variety of advantageous and beneficial properties such as clarity, shelf stability and ease of preparation. Hence, they have wide application, but especially in antiperspirants, deodorants, in perfumes as a carrier and for conditioning hair.

[0042] Our silicone elastomers, gels and pastes have uses beyond the personal care arena, including their use as a filler or insulation material for electrical cable, a soil or water barrier for in-ground stabilization or as a replacement for epoxy materials used in coil-on-plug designs in the electronics industry.

[0043] They are also useful as carrier for crosslinked silicone rubber particles. In that application, (i) they allow ease of incorporation of the particles into such silicone or organic phases as sealants, paints, coatings, greases, adhesives, antifoams and potting compounds; and (ii) they provide for modifying rheological, physical or energy absorbing properties of such phases in either their neat or finished condition.

[0044] In addition, our silicone elastomers, gels and pastes are capable of functioning as carriers for pharmaceuticals, biocides, herbicides, pesticides and other biologically active substances; and can be used to incorporate water and water-soluble substances into hydrophobic systems. Examples of some water-soluble substances are salicylic acid, glycerol, enzymes and glycolic acid.

[0045] The following examples further illustrate our invention in more detail.

Example 1

[0046] A gel was prepared using the following ingredients:

- (i) 50 g of an =SiH siloxane having an average structure represented by $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_{93}(\text{MeHSiO})_6\text{SiMe}_3$
- (ii) 260 g of decamethylcyclopentasiloxane (hereafter D5)
- (iii) 1.78 g of 1,5-hexadiene, and
- (iv) 0.60 g of Karstedt's catalyst with a Pt content of 0.52 %.

[0047] Karstedt's catalyst is a preferred platinum catalyst herein and is more fully described in US Patents 3,715,334 and 3,814,730. According to these patents, Karstedt's catalyst is a platinum divinyl tetramethyl disiloxane complex, containing 0.5-1.0 weight percent of platinum, carried in a solvent such as toluene.

[0048] A mixture of the above ingredients was stirred in a capped container and heated at 60°C . until gelation. The gel was then heated in a $65\text{--}70^\circ\text{C}$. oven for one hour. The gel was next sheared and swollen with additional D5, to form a silicone paste containing 10 wt % of an elastomer. A solution (0.5 wt % of methionine in methyl ester), 117 mg, was added (as the post cure terminating agent) and mixed with 117 g of the above silicone paste to yield a methionine content of 5 ppm in said paste. The resulting product had a viscosity of 176,000 cP (mPa·s) at 25°C . two hours after it had been prepared. One day later, the paste remained smooth and the viscosity had increased only slightly to 190,000 cP (mPa·s) at 25°C . The viscosity was measured using a Brookfield™ DV-II Viscometer having a TC-type spindle operated at a speed of 2.5 rpm (0.26 rad/s).

Example 2A - Comparative Example

5 [0049] Another gel was prepared according to Example 1, sheared and swollen in D5 to form a silicone paste containing 10 wt% elastomer. No post cure terminating agent (sulfur-containing amino acid ester) was added. The silicone paste was placed in a jar and gelled to a semisolid after 12 hours.

Example 2B - Comparative Example

10 [0050] A third gel was prepared according to Example 1, sheared and swollen in D5 to form a silicone paste containing 10 wt% elastomer. Instead of adding a sulfur containing amino acid ester as in Example 1, a solution (0.5 wt% of triphenylphosphine in ethyl acetate) 95 mg, was mixed with 117 g of the silicone paste. The triphenylphosphine content in the silicone paste was 4.1 ppm. The viscosity of the silicone paste two hours after preparation was 290,000 cP (mPa-s). One day later, the silicone paste was a soft gel that had a viscosity of 330,000 cP (mPa-s) at 25°C. The viscosity in this example was measured as in Example 1. This comparative example proves that a similar weight of methionine methyl ester from Example 1 stops post cure more effectively than triphenylphosphine.

Claims

- 20 1. A method of making a silicone elastomer comprising combining and reacting (A) a $\equiv\text{Si-H}$ containing polysiloxane of formula $\text{R}_3\text{SiO}(\text{R}'_2\text{SiO})_a(\text{R}''\text{HSiO})_b\text{SiR}_3$ and optionally a $\equiv\text{Si-H}$ containing polysiloxane of formula $\text{HR}_2\text{SiO}(\text{R}'_2\text{SiO})_c\text{SiR}_2\text{H}$ or a $\equiv\text{Si-H}$ containing polysiloxane of formula $\text{HR}_2\text{SiO}(\text{R}'_2\text{SiO})_a(\text{R}''\text{HSiO})_b\text{SiR}_2\text{H}$ where R, R' and R'' are alkyl groups of 1-6 carbon atoms; a is 0-250; b is 1-250; and c is 0-250; with (B) an alkene; (C) a platinum group metal catalyst and (D) a solvent; until said elastomer is formed by crosslinking and addition of $\equiv\text{Si-H}$ across double bonds in said alkene.
- 25 2. The method of claim 1 wherein said solvent is selected from the group consisting of (i) organic compounds, (ii) compounds containing a silicon atom, (iii) mixtures of organic compounds, (iv) mixtures of compounds containing a silicon atom, and (v) mixtures of organic compounds and compounds containing a silicon atom.
- 30 3. The method of claims 1 or 2 further including the steps of adding additional amounts of solvents to the silicone elastomer and adding an effective amount of a post cure amino acid ester terminating agent (E); and subjecting the solvent, the post cure terminating agent and the elastomer to shear force until a paste is formed.
- 35 4. The method of claim 3 in which the post cure terminating agent is methionine methyl ester, methionine ethyl ester, cysteine methyl ester, cysteine ethyl ester or cystine dimethyl ester.
- 40 5. A personal care product containing the paste obtained by the method of claim 3 or 4 selected from antiperspirants, deodorants, skin creams, skin care lotions, moisturizers, acne removers, wrinkle removers, facial cleansers, bath oils, perfumes, colognes, sachets, sunscreens, pre-shave lotions, after-shave lotions, liquid soaps, shaving soaps, shaving lathers, hair shampoos, hair conditioners, hair sprays, mousses, permanents, depilatories, cuticle coats, make-ups, color cosmetics, foundations, blushes, lipsticks, lip balms, eyeliners, mascaras, oil removers and cosmetic removers.
- 45 6. A product containing the paste obtained by the method of claim 3 or 4 and a material selected from crosslinked silicone rubber particles, pharmaceuticals, biocides, herbicides, pesticides, water and water-soluble substances.
7. Use of the personal care product of claim 5 by applying to the hair, skin or underarm.
- 50 8. A method of modifying rheological, physical or energy absorbing properties, of silicone or organic phases selected from sealants, paints, coatings, greases, adhesives, antifoams and potting compounds, comprising incorporating therein the paste of claim 5 containing crosslinked silicone rubber particles.
- 55 9. A method of filling or insulating an electrical cable comprising incorporating therein the paste obtained by the method of claim 3 or 4.
10. A method of stabilizing in-ground soil or water barriers comprising incorporating into soil the paste obtained by the method of claim 3 or 4.



European Patent
Office

EUROPEAN SEARCH REPORT

Application Number
EP 98 30 8835

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X,D	US 5 654 362 A (SCHULZ JR WILLIAM JAMES ET AL) 5 August 1997 * claims 1-18; examples 1-3 *	1,2,5-10	C08J3/09 C08G77/32
			TECHNICAL FIELDS SEARCHED (Int.Cl.6)
			C08J C08G
The present search report has been drawn up for all claims			
Place of search MUNICH		Date of completion of the search 1 February 1999	Examiner Hoffmann, K
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p>			

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
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01-02-1999

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5654362 A	05-08-1997	NONE	